Technical Bulletin

sciTEM – A loading method for water-based gold nanoparticles onto SEM and TEM supports for high-throughput characterization

Introduction

TEM and SEM measurements are commonly applied as methods for characterization of nanomaterials. However, by nature of electron microscope instrument design placing sample supports under high vacuum, the number of samples that can be measured in a given time period is very limited. Having the capability to deposit multiple samples on one TEM/SEM support and then acquire all images without breaking vacuum for each one, results in a significant increase in the number of samples that can be analyzed per measurement cycle. A piezoelectric nanoliter dispensing technology, sciTEM, is applied for high precision non-contact printing of spots onto the supports.

Results of sample loading and measurement

Samples of nanoparticles are commonly prepared in organic solvents or aqueous solutions. Due to their different surface tensions both systems require different handling when spotting the solutions onto a TEM/SEM support. Water-based nanoparticle suspensions can be handled with sciTEM to dispense sub-nanoliter droplets onto various support films without problem. However, the small drop volumes and the low surface tensions of water can results in strong sample agglomeration within the spots on the support film after evaporation of the solvent. Significant improvement of sample distribution within the spots can be obtained when support films with adjusted wetting properties are used.

An example of water-based gold nanoparticles spotted as replicates on a functionalized silicon nitride support, Figure 1. It was possible to spot a symmetric array consisting of 3x3 spots (each 250 pL) with uniform diameters of 150 µm. SEM measurements (transmission mode and in lens) show a homogeneous distribution of the particles, Figure 2. No background enhancement has been detected in the electron microscopy measurements when using surface modified support films.

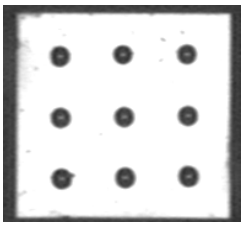


Figure 1. Spotted array of nine replicates on a 1x1 mm silicon nitride membrane.

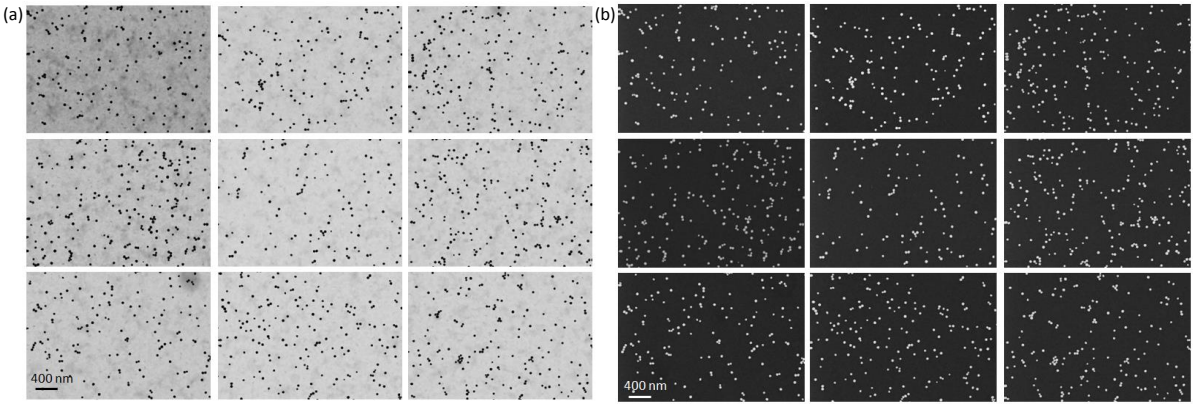
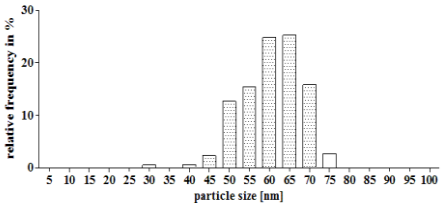


Figure 2. SEM images of bPEI stabilized gold nanoparticles on a functionalized grid with silicon nitride membrane (SIMPore), (a) transmission mode, (b) in lense-detection.



Further analysis of the images was performed to calculate diameters and size distribution of gold nanoparticles, Figure 3. The typical particle diameters of the investigated sample range from 50 to 70 nm.

Figure 3. Size distribution of bPEI gold nanoparticles printed on a functionalized silicon nitride membrane.

Summary

The sciTEM technology can be used to load support films with multiple samples of nanoparticles. Using surface functionalized silicon nitride supports grids with optimized wetting properties can minimize agglomerations of water-based gold nanoparticle dispersions.

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